



# Durability Evaluation of a Thin Film Sensor System With Enhanced Lead Wire Attachments on SiC/SiC Ceramic Matrix Composites

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## **ABSTRACT**

An advanced thin film sensor system instrumented on silicon carbide (SiC) fiber reinforced SiC matrix ceramic matrix composites (SiC/SiC CMCs), was evaluated in a Mach 0.3 burner rig in order to determine its durability to monitor material/component surface temperature in harsh environments. The sensor system included thermocouples in a thin film form (5  $\mu\text{m}$  thick), fine lead wires (75  $\mu\text{m}$  diameter), and the bonds between these wires and the thin films. Other critical components of the overall system were the heavy, swaged lead wire cable (500  $\mu\text{m}$  diameter) that contained the fine lead wires and was connected to the temperature readout, and ceramic attachments which were bonded onto the CMCs for the purpose of securing the lead wire cables. The newly developed ceramic attachment features a combination of hoops made of monolithic SiC or SiC/SiC CMC (which are joined to the test article) and high temperature ceramic cement. Two instrumented CMC panels were tested in a burner rig for a total of 40 cycles to 1150 °C (2100 °F). A cycle consisted of rapid heating to 1150 °C (2100 °F), a 5 minute hold at 1150 °C (2100 °F), and then cooling down to room temperature in 2 minutes. The thin film sensor systems provided repeatable temperature measurements for a maximum of 25 thermal cycles. Two of the monolithic SiC hoops debonded during the sensor fabrication process and two of the SiC/SiC CMC hoops failed during testing. The hoops filled with ceramic cement, however, showed no sign of detachment after 40 thermal cycle test. The primary failure mechanism of this sensor system was the loss of the fine lead wire-to-thin film connection, which either due to detachment of the fine lead wires from the thin film thermocouples or breakage of the fine wire.

## **INTRODUCTION**

In recent years, there has been a surge of interest in the fiber reinforced ceramic matrix composite (CMCs) for their potential for extreme high temperature applications in the aeronautics, chemical, electronics, energy, nuclear, and transportation industries. Some of the potential applications include aircraft engine components, components for reusable and expendable launch vehicles (RLVs and ELVs), radiant heater tubes, heat exchangers and recuperators, and components for land based turbines for power generation. Thorough characterization of CMC properties, including testing under simulated or actual operating harsh conditions, is a high priority for research programs involved in the development. This requires the development of advanced, robust minimally intrusive measurement systems such as thin film sensors. The thin film sensors are sputter deposited directly onto the surface of the article that is to be tested. They have a thickness on the order of a few micrometers ( $\mu\text{m}$ ), which is many orders of magnitude thinner than the conventional sensors (in the wire form). Thin film sensors add negligible mass to the surface and create minimal disturbance of the gas flow over the surface. Consequently, thin film sensors have minimal impact on the thermal, strain and vibration patterns that exist in the operating environment and provide more accurate readings with

a faster response time. In aeronautics and aerospace applications, where stress and temperature gradients are high and aerodynamic effects need to be minimized, sensors in a thin film form provide a minimally intrusive means of monitoring the surface parameters, such as strain or temperature, in hostile environments [1-5].

The process for fabricating thin film sensors can be tailored to provide very good adhesion to ceramics or CMCs. However, poor adhesion of 1) the fine lead wires to the thin films and 2) the lead wire cables to the test articles often prevents successful transmission of the signals from the sensors. When instrumenting metallic components, the attachment of the lead wire cables can be easily done by spot welding directly onto the test articles. The attachment of wires on non-metallic components such as ceramics or CMCs is very difficult, and has been a major challenge to researchers over the years. The attachment of lead wire cables with commercially available ceramic cements fails to provide sufficient adhesion at high temperatures. These ceramic-based cements often crack and debond, which leads to the detachment of the lead wires from the test articles. In harsh testing environments, i.e., exposure to high temperature and high pressure, the durability or lifetime of the thin film sensors is therefore somewhat limited.

In order to address the lead wire attachment problems, an affordable, robust ceramic joining technology named *ARCJoinT* was used to bond monolithic SiC or SiC/SiC composite hoops on SiC-based subcomponents for the purpose of securing sensor lead wires in place [6]. The *ARCJoinT* approach developed at the NASA Glenn Research Center (GRC) for the joining of SiC-based ceramics and fiber reinforced composites has been shown to produce joints with tailorable thickness and good high temperature strength (265 MPa at temperatures up to 1350 °C in air) [7-13]. This joining technique is attractive since the thermomechanical properties of the joint interlayer can be tailored to be very close to those of the SiC-based substrate materials. Therefore, this novel attachment approach is being evaluated to determine its potential for enhancing the durability of the high temperature thin film sensor systems.

## EXPERIMENTAL PROCEDURES

### Instrumentation of Composite Specimens

Two melt infiltrated (MI) HiNicalon SiC fiber reinforced SiC matrix composite panels (4 cm wide, 19 cm long, and 2 mm thick) were instrumented as described below: 1) SiC monolithic or SiC/SiC composite hoops were joined to these panels using a *ARCJoinT* process; 2) the panels were instrumented with thin film thermocouples; and 3) lead wire cables were threaded through the hoops and further secured with ceramic cement. Finally, the fine lead wires were welded to the thin films. The instrumented panels are shown in Fig. 1.

#### *1) Fabrication and Joining of SiC-Based Hoops*

Hoops made of two types of materials, monolithic reaction formed SiC and SiC/SiC composite, were used as attachments. The hoops were fabricated from either 1) a carbon tube (4.9 mm o.d., with a wall thickness of 1.6 mm) that was ground to form the desired cross section (hoop shape) and then reacted with silicon to form a reaction bonded SiC piece or 2) a special CVI SiC/SiC composite panel that had been fabricated with tooling that created hoop-shaped ridges in the panel. The monolithic SiC hoops were semicircular. Each hoop was 1 mm wide and 3.1 mm high (hole was 1.5 mm high). The SiC/SiC hoops were elongated rather than semicircular. Each SiC/SiC composite hoop was 2 mm wide, 1 to 1.5 mm thick, and 12 mm long, with an opening that was 5 mm wide and 2 mm high (semi-oval-shaped hole), Fig. 2. The intent was to use hoops that had an opening just slightly larger than the diameter of the lead wire cable, in order to ensure that the hoops would secure the lead wires and prevent them from moving. Such hoops will be required to minimize the disruption of high velocity gas flow under simulated engine conditions.

Two columns of hoops were joined on each CMC panel (Fig. 1). SiC hoops were joined to one of the panels (bottom panel on Fig 1), and SiC/SiC hoops were joined to the other panel (top panel on Fig 1). A carbonaceous mixture was first applied to the joint area between the hoop and the panel. This was followed by curing at 110 to 120 °C for 10 to 20 minutes [6]. During curing, a moderately strong bond was developed between the hoops and the SiC/SiC CMC test panels. In the next step, silicon paste was applied to the joint regions, and the test panels were heated up to 1425 °C for 10 minutes. The silicon paste became molten and reacted with the carbonaceous material to form a joint consisting of silicon carbide with a minor amount of silicon. The average joint thickness obtained was not determined; however, those observed in our previous attachment study were approximately 150 μm thick [6].

## **2) Fabrication of Thin Film Sensors**

The fabrication of the thin film sensors was performed in a clean room to minimize possible contamination. The CMC panel was first grit-blasted with alumina grit to roughen the surface, and then heat-treated at 1200 °C for 50 hr to form an approximately 2 to 3  $\mu\text{m}$  thick layer of silicon dioxide on the surface. A layer of alumina approximately 2 to 4  $\mu\text{m}$  thick was then deposited at 800 °C to form the insulating base coat. Alumina was used because it has the highest resistivity of all the dielectric materials. Two side-by-side type-R thin film thermocouples were patterned onto this alumina film with stenciled shadow masks during sputter deposition of the platinum (Pt) and platinum-13 wt % rhodium (Pt-13Rh) thin films as shown in Fig. 1. Sputter depositions were performed under Magnetron mode with a power density of 2.5  $\text{W}\cdot\text{cm}^{-2}$ . Approximately 5  $\mu\text{m}$  thick Pt and Pt-13Rh films were prepared, with the substrate heated at approximately 200 to 250 °C during the deposition process. The heat was obtained from an adjacent heater that was maintained at 400 °C during the deposition process. The instrumented CMC panel was then heat treated at 1100 °C for 10 hr to anneal and stabilize the thin film sensor.

## **3) Lead Wire Routing and Attachment**

Five hundred-micrometer (20-mil) diameter IN600 swage cable was used as the heavy lead wire in this study. The cable contained two 75  $\mu\text{m}$  diameter conductors made of Pt-13Rh and Pt, respectively, with magnesium oxide (MgO) insulation. Swaged cables were slipped through each column of the hoops. Four hoops on each panel with the excess space between the cable and the hoop were filled with high temperature ceramic cement to prevent the cable from moving. The 75  $\mu\text{m}$  diameter fine wires were attached to the thin films via the parallel gap welding process. Lead wires made of Pt-13Rh and Pt were welded to the Pt-13Rh and Pt thin film thermocouple elements, respectively.

## **Testing Conditions**

The instrumented CMC panels were tested in a Mach 0.3 burner rig, whose configuration is shown in Fig. 3. The combustor burns jet fuel and air in controlled ratios. It produces a flame (having a Mach 0.3 velocity) from the exhaust nozzle, which impinges on the test specimen. In this study, each panel was positioned so that an approximately 2.54 cm (1 in.) diameter hot zone was centered on the first row of the hoops, Fig. 4. The jet fuel to air ratio of the burner was controlled so that the panel would reach a maximum of 1150 °C (2100 °F) at the center of the hot zone. Optical pyrometry was used to verify that this temperature (1150 °C) was obtained. A total of 40 thermal cycles were conducted to evaluate the durability of sensor systems. A thermal cycle consisted of rapid heating to 1150 °C, a 5 min hold at 1150 °C, and then cooling down to room temperature in 2 min. This rapid cooling was achieved with forced cooling by flowing service air over the panel and providing thermal shock. Again, the system elements evaluated during this testing included the thin films, the fine lead wires and their attachment to the thin film, joined SiC-based hoops, the lead wire cables, and the high temperature ceramic cement, as shown in Fig. 5.

## **RESULTS AND DISCUSSION**

All of the SiC/SiC composite hoops survived the thin film sensor fabrication process, which included surface preparation by grit blasting, heat treatments at 1200 °C for 50 hr in order to form silicon dioxide, physical vapor deposition (described earlier), and 10 hr annealing in air at 1100 °C. However, two SiC monolithic hoops broke during the grit blasting process (fourth hoop on the upper side of the bottom panel and fifth on the lower side, Fig. 1)

### **CMC Panel with SiC/SiC Composite Hoops**

The thin film thermocouple on the lower side of the panel failed during the first cycle to 1150 °C due to the 75  $\mu\text{m}$ -lead wires detaching from the thin film. Testing continued after repair of the welds. During the second cycle, the thermocouple on the upper side failed, while the thermocouple on the lower side continued to function through 25 cycles. The variation in CMC temperature that was monitored by the bottom thermocouple is shown in Fig. 6. The data presented are only for thermal cycles 18 to 25. Note that the center of the hot zone, which was only 2.54 cm (1 in.) in diameter, was focused on the first row of the hoops. The thin film thermocouple, with its junction outside this hot zone, was therefore expected to read a temperature lower than that of the center of the zone. In this case, the thin film thermocouple read 1010 °C (1850 °F) in comparison with the 1150 °C (2100 °F) maximum temperature in the center area. Note that the thermocouple provided repeatable readings between thermal cycles.

Examination of the instrumented panel following the 25<sup>th</sup> cycle, as shown in Fig. 7, revealed that each of the thin films was still intact and continuous. The fine 75  $\mu\text{m}$  diameter wires, however, were gone (see no. 1, Fig. 7). The two hoops in the second row, which had no cement “filling” were gone (no. 2, Fig. 7), and it appeared that the loss of the hoop on the top may have resulted in the breaking of the swaged lead wire cable (no. 3, Fig. 7). The hoops with cement filling all survived the durability test, however, some cement placed by itself on the surface was gone (no. 4, Fig. 7). The panels were put back in the burner rig and subjected to another 15 cycles of testing, but no more data was collected due to the loss of the fine lead wires. Post-testing examination following the 40<sup>th</sup> cycle revealed that all of the thin films were still intact, except a small piece of Pt-13Rh film delaminated from the panel (no. 5, Fig. 7). No additional losses of the hoops were observed.

#### **CMC Panel with SiC Monolithic Hoops**

The temperatures measured by one of the thin film sensor systems on this panel during the burner rig testing are shown in Fig. 8. Note that the thin film thermocouple measured a maximum temperature of 1000 °C (1800 °F), which was lower than the expected 1150 °C (2100 °F). This is again due to the location of the thermocouples’ junctions, which were outside the hot zone. The thermocouple on the bottom failed during the second thermal cycle and the thermocouple on the top failed during the 8<sup>th</sup> thermal cycle. The data shown in Fig. 8 are from cycles 2 through 8. It was determined that failure occurred at the fine lead wire to thin film joints (see no. 1, Fig. 8). No data was taken after the eighth cycle due to the loss of the lead wires. The Pt-13Rh thin film of the top thermocouple started to delaminate at the junction area after the 15<sup>th</sup> cycle (no. 2, Fig. 9). At the end of the 40<sup>th</sup> cycle, the Pt-13Rh thin film legs of both thermocouples had some delamination. Although, two SiC monolithic hoops broke during the sensor preparation process, all of the remaining hoops survived the burner rig test.

#### **CONCLUSIONS**

Measurement of the surface parameters (such as strain or temperature) of advanced materials such as CMCs in hostile environments has been a difficult task due to the poor adhesion of the measurement systems. Improvements in the reliability and durability of the constituents of sensors in the high temperature, high-pressure, and high velocity gas stream environments are therefore needed. Thin film sensors can be tailored to have very good adhesion to ceramics or CMCs. However, the poor adhesion of the lead wires to both the thin films and the test articles often prevents successful transmission of the signals from the thin film sensors.

A robust ceramic joining technology, ARCJoinT, was used to bond monolithic SiC or SiC/SiC composite hoops on SiC/SiC CMC panels that were subsequently instrumented with thin film thermocouples. The hoops served as attachments for securing the sensor heavy lead wires. SiC or SiC/SiC hoops filled with ceramic cement proved to have excellent strength; they secured the lead wire cables and prevented them from moving during 40 thermal cycles to 1150 °C (2100 °F). The thin film thermocouples, combined with this enhanced lead wire attachment approach, provided readings for up to 25 thermal cycles during testing in a Mach 0.3 burner rig. The major failure mechanism was the loss of the lead wire-to-thin film thermocouple connection. Improving the durability of this portion of the sensor system will be the subject of a future study.

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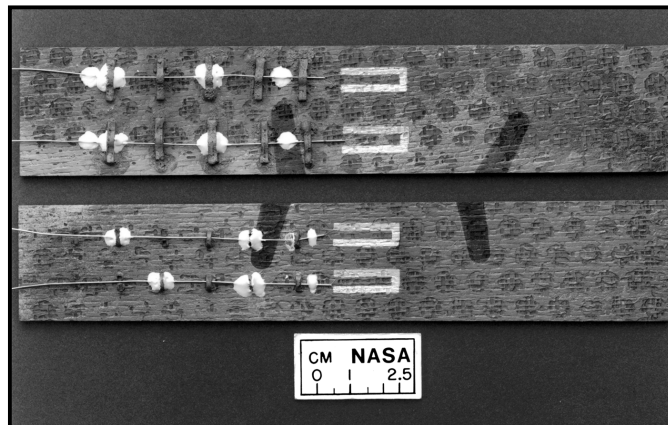


Fig. 1.—SiC/SiC CMC panels instrumented with thin film thermocouples. Lead wire cables were secured with joined SiC/SiC composite hoops (top) or SiC (bottom) monolithic hoops and refractory ceramic cement.

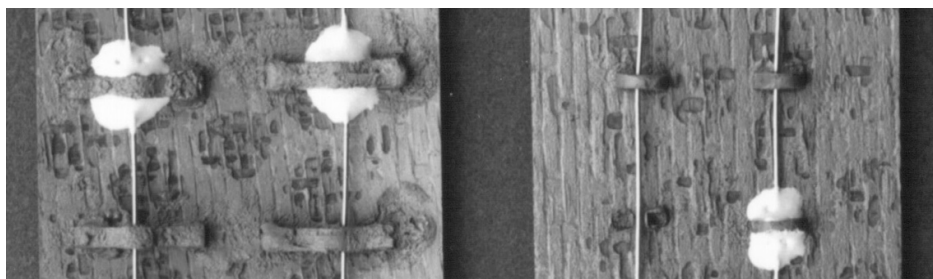


Fig. 2.—Hoops made of SiC/SiC CMC (left) or monolithic SiC (right) joined to the panels for securing lead wires.

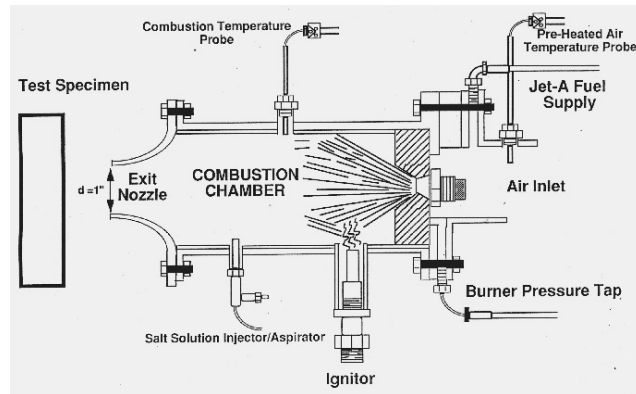


Fig. 3.—Schematic of the Mach 0.3 burner rig that was used to test the instrumented panels.

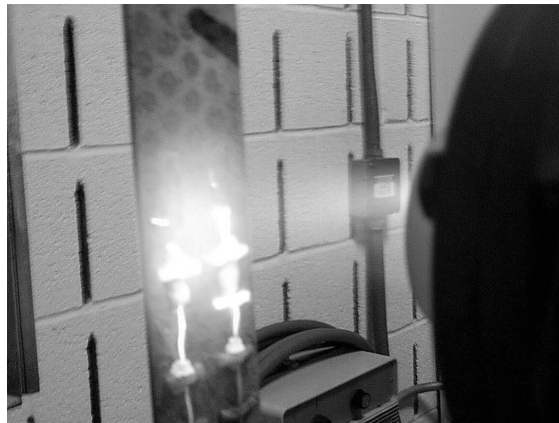


Fig. 4.—Burner rig testing of a CMC panel with SiC/SiC composite hoops.

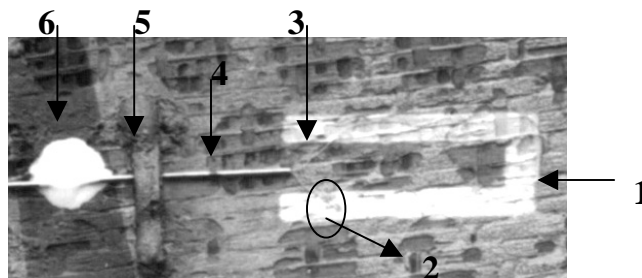


Fig. 5.—A closed-up view of a thin film sensor system which include 1) thin film; 2) thin film-to-fine lead wire joint; 3) fine lead wire; 4) swage cable; 5) SiC-based hoops and 6) ceramic cement.

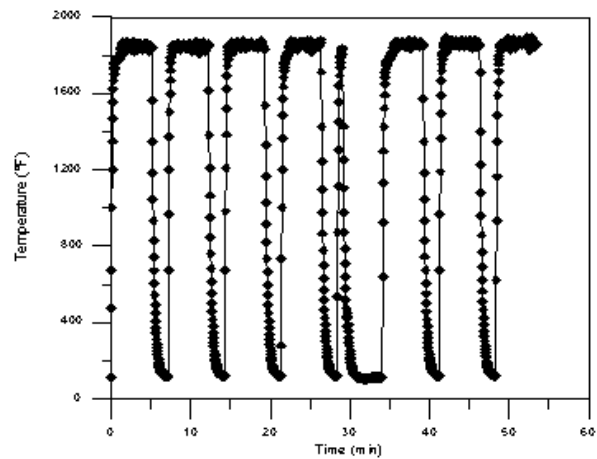


Fig. 6.—Surface temperature of a SiC/SiC CMC measured by a thin film thermocouple. The lead wire cable was secured with SiC/SiC composite hoops and ceramic cement.

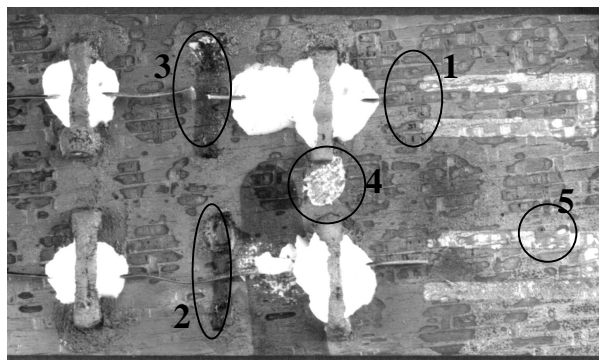


Fig. 7.—SiC/SiC CMC panel with SiC/SiC composite hoops after 40 thermal cycles to 1050 °C test in a Mach 0.3 burner rig.

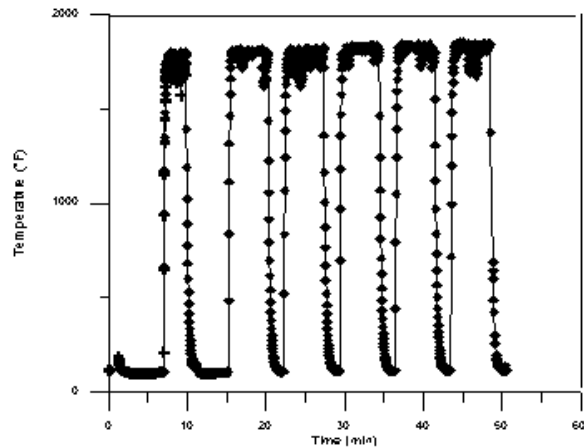


Fig. 8.—Surface temperature of a SiC/SiC CMC measured by a thin film thermocouple. The lead wire cable was secured with SiC hoops and ceramic cement.

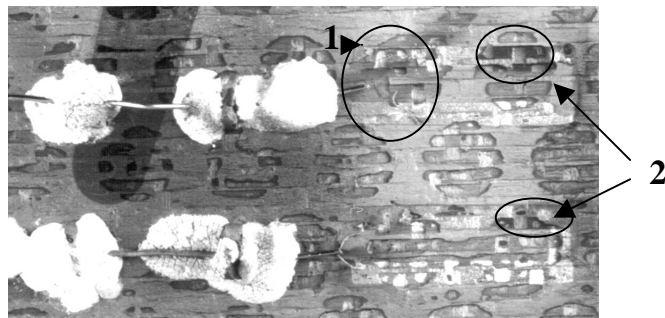


Fig. 9.—SiC/SiC CMC panel with SiC hoops after 40 thermal cycles to 1050°C test in a Mach 0.3 burner rig.

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